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EIDGENÖSSISCHES AMT FÜR GEISTIGES EIGENTUM

PATENTSCHRIFT

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Klasse 24a

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Master patent

HAUPTPATENT

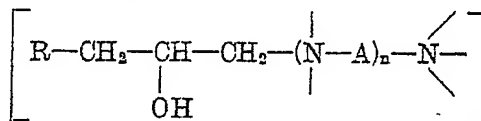
Sandoz AG., Basel (Schweiz)

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Verfahren zur Herstellung beständiger Färbe- und Abziehbäder "Process for preparation of stable dye stripping baths"

Die vorliegende Erfindung betrifft ein Verfahren zur Herstellung beständiger Färbe- und Abziehbäder mit das Auf- oder Wiederaufziehen der Farbstoffe verzögernder Wir-

kung, welches dadurch gekennzeichnet ist, daß man diesen Bädern eine Verbindung zu- setzt, welche ein positives Ion von der Formel:

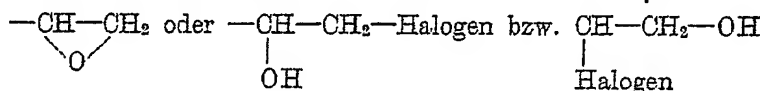


bilden kann, worin R einen mindestens 8 Kohlenstoffatome enthaltenden Rest, der außer Kohlenstoffatomen nur noch die Atome des Wasserstoffes und Sauerstoffes enthält und der durch ein Sauerstoffatom mit dem restlichen Ion-Teil verbunden ist, A Alkylreste mit 2—4 Kohlenstoffatomen und n eine ganze Zahl, die größer als 0 ist, bedeuten.

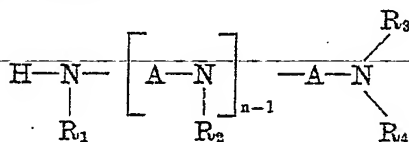
Verbindungen, welche obengenanntes Ion bilden können, sind z. B. solche, die durch Umsetzung von Verbindungen der Formel:



worin R die obengenannte Bedeutung besitzt und worin X die Gruppe:

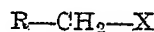


bedeutet, mit Polyaminen der Formel:



worin R₁, R₂, R₃ und R₄ Wasserstoffatome, Alkyl-, Aralkyl-, Oxalkyl- und Polyglykol-ätherreste, A Alkylreste mit 2—4 Kohlenstoffatomen und n eine ganze Zahl, die größer als 0 ist, bedeuten, erhältlich sind, oder solche, die aus diesen durch Alkylierung gewonnen werden.

Geeignete Verbindungen der Formel

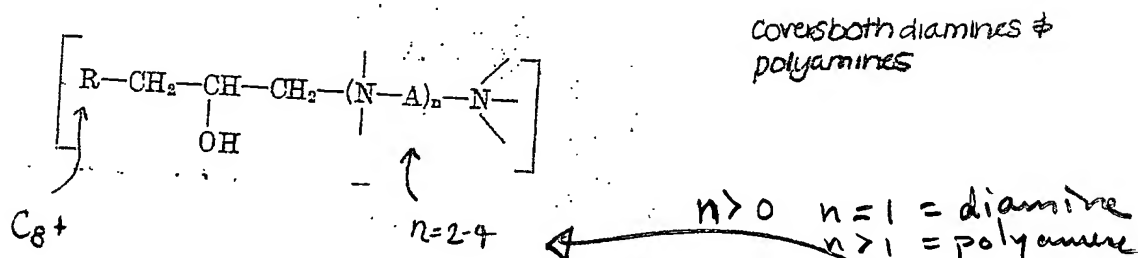


sind z. B. Octyl-, Dodecyl-, Hexadecyl-, Octadecyl-, Octadecenyl-oxy-propenoxyde, Alkylphenoxy-propenoxyde, ferner Verbindungen der Formel:

Swiss CH 313159

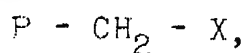
Process for preparation of stable dye and stripper baths.--

The present invention concerns a process for preparation of stable/permanent dye and stripper baths with an effect retarding the absorption or reabsorption of the dyestuff, which is characterized in that one adds to these baths a compound which can form a positive ion of the formula

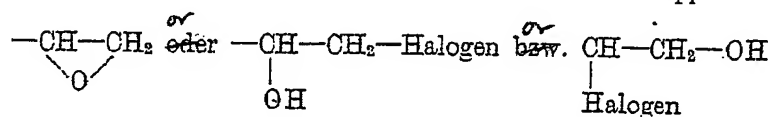


where R is a radical residue containing at least 8 carbon atoms which aside from carbon atoms only contains atoms of hydrogen and oxygen and which is linked by an oxygen atom with the residual ion part, A means alkylene residues with 2-4 carbon atoms, and n is an integer greater than 0.

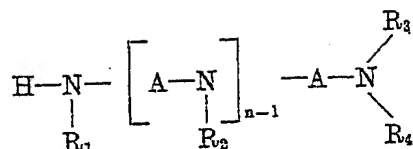
Compounds which can form the aforesaid ion are e.g. ones such as are obtained by conversion of compounds of the formula



where R has the aforesaid meaning and where X means the group:

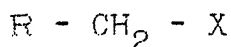


with polyamines of the formula:

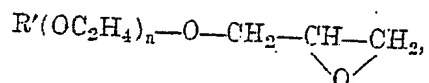


wherein R_1 , R_2 , R_3 and R_4 are hydrogen atoms, alkyl, aralkyl, oxalkyl and polyglycol ether residues, A means alkylene residues with 2-4 carbon atoms and n is an integer greater than 0, or ones that can be obtained therefrom by alkylation.

Suitable compounds of the formula



are e.g. octyl, dodecyl, hexadecyl, octadecyl, octadecenyl-oxv-propene oxides, alkyl-phenoxv-propene oxides, and, further, compounds of the formula:



wherein n is an integer and R' is an arbitrary carbon residue containing at least 8 carbon atoms or the halogen hydrines corresponding to the above epoxides.

Suitable polyamines are e.g. **DETA** diethylene triamine, dipropylene triamine, polyethylene and/or polypropylene polyamines, methyl aminoethyl amine, 3-isopropylamino-propylamine-1, 3,3-dimethyl aminopropylamine-1.

The conversion products, should they still exhibit alkylatable nitrogen atoms, can be alkylated or peralkylated (quaternized) by treatment with alkylation agents such as ethylene oxide, dimethyl or diethyl sulfate, chloroacetic acid, chloroxv propane sulfonic acid, benzyl chloride, etc.

The conversion of the aforementioned components is conducted expediently at a high temperature, e.g. between 40 and 160°. The conversion products are oily to viscous products, very ^{easily} ~~slightly~~ soluble in water, which can be used in neutral, acidic or alkaline baths. They are suitable for preparation of stable dye and stripper baths, where they can be used "as-is" or in a mixture with appropriate additives.

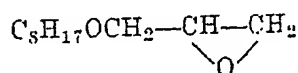
Dye or stripper baths containing compounds of this nature in solution or in an appropriate dispersion have a good equalization, penetrating or stripper effect for direct, acidic, basic chrome and vat (copper) dyes.

The compounds of the cited nature are added to the dye and stripper baths in amounts of around 0.02-0.1 g/liter and, as such, ^{or} applied along with other equalization agents, exert a retardation effect on the absorption of the dyes, leading to equal colorations.

20-100 ppm

Below we will mention the preparation of some few products and then illustrate/discuss their applicability as per invention based on examples. They are obtained by conversion of the following compounds in the stated mol ratio and in the stated sequence:

A. 1 mol n-octyloxypropeneoxide

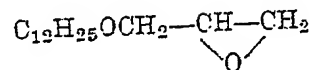


is combined with 2 mol diethylenetriamine and brought to reaction at 100-120°. After that, the non-converted diethylene triamine is distilled off in a vacuum, the residue is brought to conversion with 8 mol ethylene oxide at 100° and then treated at 50-60° with 3 mol dimethyl sulfate. The heating-up is continued for 2 h to 80-85°, after which all the dimethyl sulfate is consumed. One obtains a brownish, viscous, slightly water-soluble mass.

intermediate

In similar fashion one obtains the following products:

B. 1 mol n-dodecylpropylene oxide



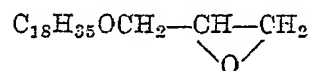
1 mol diethylene triamine (after distilling off the excess of 1 mol)

8.5 mol ethylene oxide

3 mol dimethyl sulfate.

Brownish, gelatinous, easily water-soluble mass.

C1. 1 mol oleyloxy propene oxide



1 mol diethylene triamine (after distilling off the excess of 1 mol)

8.5 mol ethylene oxide.

C2. 1 mol product C1. + 3 mol dimethyl sulfate

Easily water-soluble product.

D.

1 mol isooctyl phenoxy propene oxide

1 mol diethylene triamine (after distilling off the excess of 1 mol)

7 mol ethylene oxide

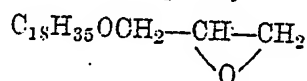
3 mol dimethyl sulfate

Brownish, readily water-soluble gelatinous/colloidal paste.

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E.

1 mol oleyloxy propene oxide



1 mol diethylene triamine (after distilling off the excess of 1 mol)

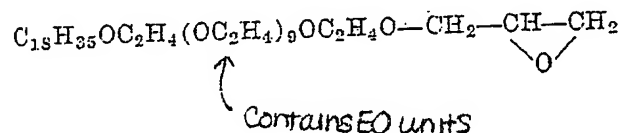
8.5 mol ethylene oxide

1.5 mol benzyl chloride

Brownish, thick-flowing/viscous, readily soluble oil.

* (F.)

1 mol of the compound



1 mol diethylene triamine or triethylene triamine or tetraethylene pentamine.

Brownish, thick-flowing/viscous oil, readily soluble in water.

G.

1 mol oleyloxy propene oxide

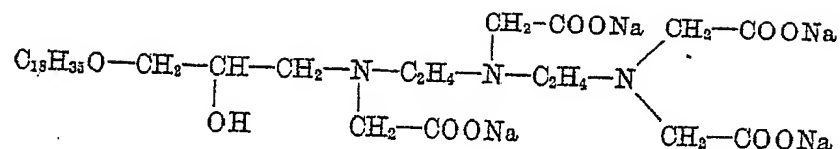
1 mol diethylene triamine (after distilling off the excess of 1 mol)

4 mol monochloroacetic acid as a 50 o/o solution at 40-50°

4 mol caustic soda as a 38 o/o aqueous solution at 60-70°

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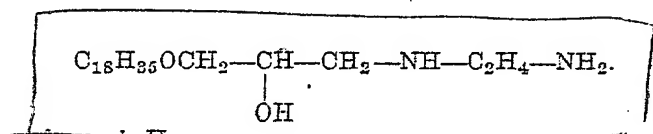
Bright powder, essentially mainly:



(H.)

* 1 mol oleovloxy propene oxide
1 mol ethylene diamine (after distilling off the excess of 1 mol)

Thick-flowing/viscous, brownish oil, essentially mainly:



I.

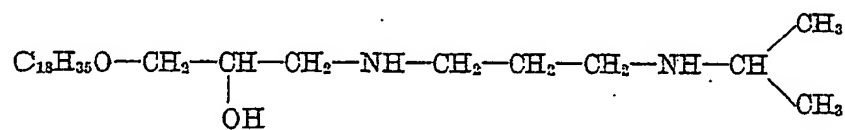
1 mol of product per H
5 mol ethylene oxide

Brownish, viscous, readily water-soluble oil.

K.

1 mol oleovloxy propene oxide
1 mol 3-isopropylamino-propylamine-1

Brownish, thick-flowing/viscous oil, essentially mainly:



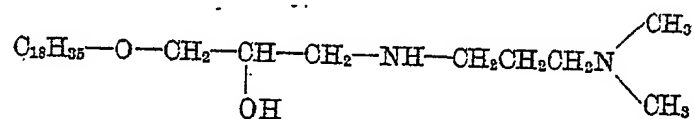
L.

- 1 mol product as in point K
- 2 mol ethylene oxide

M.

- 1 mol oleyloxypropene oxide
- 1 mol 3- dimethylamino-propylamine-1

Brownish product, essentially mainly:



N.

- 1 mol N-octadecenyl-oxypropene oxide
- 1.2 mol diethylene triamine
- 1 mol dimethyl sulfate
- (continued next page)

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1.2 mol caustic soda (as a 30 o/o solution)
4.2 mol ethylene oxide

Brown, thick-flowing/viscous, readily water-soluble oil.

O.

1 mol octadecenyl-oxypropene oxide
1.2 mol diethylene triamine
7 mol ethylene oxide
2 mol epichlorohydrine

Brown, viscous, readily water-soluble paste.

P.

2 mol octadecenyl-oxy-propene oxide
1 mol tetraethylene pentamine
4.5 mol ethylene oxide

Brownish, water-soluble oil.

Q.

1 mol octadecenyl-oxy-propene oxide
1.2 mol diethylene triamine
8.5 mol ethylene oxide
1 mol pulverized caustic soda
1 mol monochloroacetic acid sodium

Brownish, viscous, readily water-soluble paste.

R.

1 mol diamylphenoxypropeneoxide
1.5 mol diethylene triamine
7 mol ethylene oxide
3 mol dimethyl sulfate.

Water-soluble product.

S.

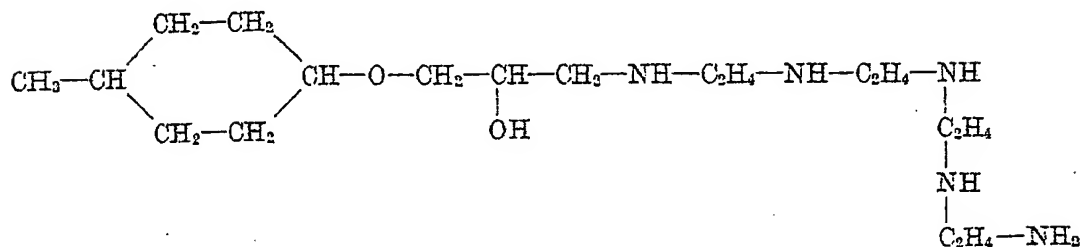
- 1 mol octadecanyl-oxypropeneoxide
- 1 mol 3-dimethylaminopropylamine
- 2 mol glycerine monochlorohydrine

Water-soluble, brown oil.

T.

- 1 mol methylcyclohexyloxypropeneoxide
- 1.2 mol tetraethylene pentamine.

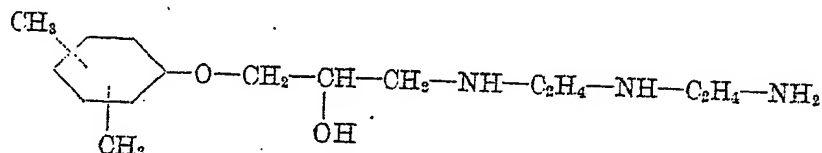
Brownish, readily water-soluble oil, essentially mainly:



U.

- 1 mol xylenyloxypropene oxide
- 1.1 mol diethylene triamine

Thick-flowing/viscous oil, essentially mainly:

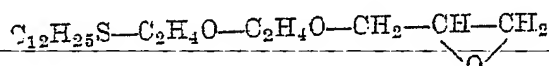


V.

- 1 mol xylenyloxypropene oxide
- 1.1 mol of a polyethylene polyamine with boiling point $> 206^{\circ}$.

W.

1 mol of



1.2 mol diethylene triamine

Brownish, thick-flowing/viscous, water-soluble oil.

Example 1.--

To prepare a stable dye bath one added to the bath per liter 0.4 g azorubinol 3GS (Schultz Farbstofftabellen, page 28, 1932), 0.8 g sulfuric acid (96 o/o), 2 g of mirabilite, 0.05 g of the product produced per C1, and 0.1 g of oleylpolyglycol ethers with 20 etheneoxy groups in the molecule.

With the aid of such a thusly produced dye bath, one can perfectly color wools in the ~~usual~~ usual way (float ratio 1:50, duration 45 min, at boiling temperature), whilst a corresponding dye bath not containing any product as per C1 unevenly dyes the same wool.

In a bath of the cited composition, one can also dye with good penetration even difficulty penetratable goods such as wool felts.

Dyestuff baths with similar products can be obtained if the product per C1 is replaced by the products F, H, M, N, O, P, Q, T, U, V and W.

Example 2.--

A patent vat was prepared as follows:

1 g of sandothrenol 3B (Schultz Farbstofftabellen, II volume, p. 128, 7th edition 1934) is vatted in a solution consisting of 95 cm³ water, 1 g of a highly sulfonated 25 o/o sulfonate, 3 cm³ of soda lye of 36° Be and 2 g sodium hydrosulfite for 15 min at 80 °C.

To prepare a vatting one adds 10 cm^3 of the thusly prepared stem vatting to 1 liter of a blind vat which contains 15 cm^3 of soda lye of 36°Be , 5 g sodium hydrosulfite, 0.025 g of the product attained by C2 and 0.025 g of the oleylpolyglycolether ethers with 20 etheneoxy groups in the molecule.

With the thusly obtained vatting one can uniformly color a boiled-down, very absorptive cotton-creton tissue at at most 50° in the float ratio of 1:40, which is not at all the case when the blind vat does not contain the product per C2.

Equally effective vattings can be obtained if one uses products per A, B, C, D or E instead of the product C2.

Example 3.--

To prepare a blind vatting one dissolves in one liter of water 15 cm^3 of soda lye 36°Be , 5 g sodium hydrosulfite, 0.45 g of the product per C2 and 0.55 g of oleylpolyglycol-ether with 20 etheneoxy groups in the molecule.

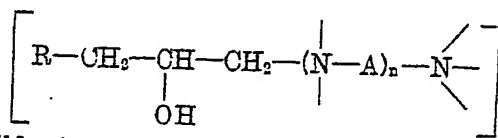
With a blind vat of this nature, one can extensively strip an un-uniformly dyed and spottily piece of cotton-cretonne with sandothrenviolett N4R (Schlutz Farbstofftabellen, second volume, 7 edition, p. 127, 1974) by means of a 30 min treatment in the float ratio of 1:40 at $95-100^\circ$, which is not the case if the blind vat does not contain the auxiliary/adjuvant obtained via C2.

A blind vat for whose preparation instead of the C2 product one uses on per G, R or S, has similar properties as the ones described above.

Instead of the oleylpolyglycolether mentioned in Examples 1, 2 and 3 one can add to the dye or stripping baths similarly effective alkyl or alkylphenyl polyglycol ethers.

Patent Claim.--

Process for preparation of stable/resistant dye or stripping baths with an effect retarding the absorption or reabsorption of the colors, characterized in that one adds to the dye or stripper baths a compound which can form a positive ion of the formula



where R is a residue containing at least 8 carbon atoms, which aside from carbon only contains atoms of hydrogen and oxygen, and which is bonded with the rest of the ion part by an oxygen atom, A means alkylene residues with 2 to 4 carbon atoms, n is an integer greater than 0.

Sandoz AG
